

## Research Article

# Evaluation of the Pozzolanic Activity of Sewage Sludge Ash

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Raw sewage sludge was characterized by XRD, FTIR, SEM, and TGA techniques and incinerated in temperature range 650–950°C for 2 h. The effect of incineration temperature on the microstructure and pozzolanic activity of the resultant ash was investigated by techniques mentioned above as well as Chapelle test. It was concluded that incineration of sewage sludge affects the microstructure and pozzolanic activity of the resultant ash. During incineration at temperatures lower than 800°C, amorphous silica captures fixed carbon resulting from incomplete combustion conditions whereas at higher temperatures crystallization of amorphous silica was enhanced. Hydration products formed from hydrothermal treatment of silica fume with lime is amorphous whereas that of sewage sludge ash is fibrous. Hence, incineration of sewage sludge ash must be optimized at 800°C to preserve the pozzolanic activity of the resultant ash.

## 1. Introduction

Sewage is the collection of wastewater effluents from domestic, hospital, commercial, industrial establishments, and rain water. The objective of sewage treatment is to produce treated sewage water and sewage sludge suitable for safe discharge into the environment or reuse [1]. The most common treatment options for sewage sludge include anaerobic digestion, aerobic digestion, and composting. Choice of the treatment method depends on the amount of sludge and other site-specific conditions [2]. Sewage sludge tends to accumulate heavy metals existing in the wastewater. The composition of sewage sludge and its content of heavy metals vary widely depending on the sludge origins and treatment options [3]. In past decades, sewage sludge was primarily disposed to landfills and seawaters [4]. Space limitations on existing landfills and increasing environmental concerns such as groundwater pollution from landfill leachate, odor emission and soil contamination have prompted the investigation of alternative disposal routes [5]. Sewage sludge has been used in agriculture as fertilizer and soil amendment [6]. However heavy metals such as Zn, Cu, Ni, Cd, Pb, Hg, and Cr are principal elements restricting the use of sludge for agricultural purposes [3]. The solidification/stabilization of sewage sludge in cementitious matrix has been evaluated [7]. Sewage sludge adversely affects durability of concrete,

mainly due to the organic material which retards setting and heavy metals which interfere with the hardening reactions [8]. Hence, sewage sludge addition in concrete was limited to 10 wt.% and the solidified concrete was recommended for use in certain very specific applications [9]. Sewage sludge also has been used in production of clay bricks [10].

Application of sewage sludge without appropriate management might have adverse impacts on human health and the environment [11]. Legislative barriers for sewage sludge disposal options make incineration is the most attractive disposal method [12]. The advantages of sewage sludge incineration summarized as follows:

- (i) large reduction of sludge volume by about 90% [13],
- (ii) thermal destruction of toxic organic compounds [14],
- (iii) recovery of much of the energy in the sewage sludge [15],
- (iv) minimization of odor emission,
- (v) making further management of sludge ash an easier task.

Sewage sludge ash has been used as an additive in production of construction materials [16], mortars [17], concrete [18], brick [19], asphalt paving mixes [20], aerated

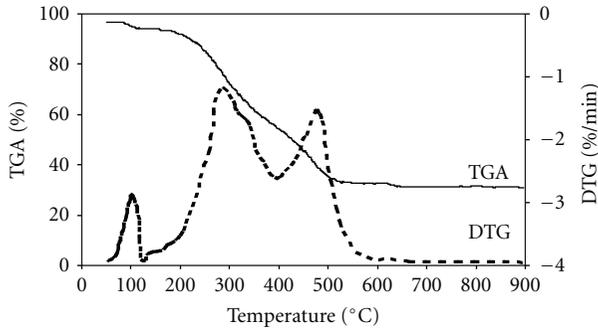


FIGURE 1: The TGA and DTG thermograms of raw sewage sludge.

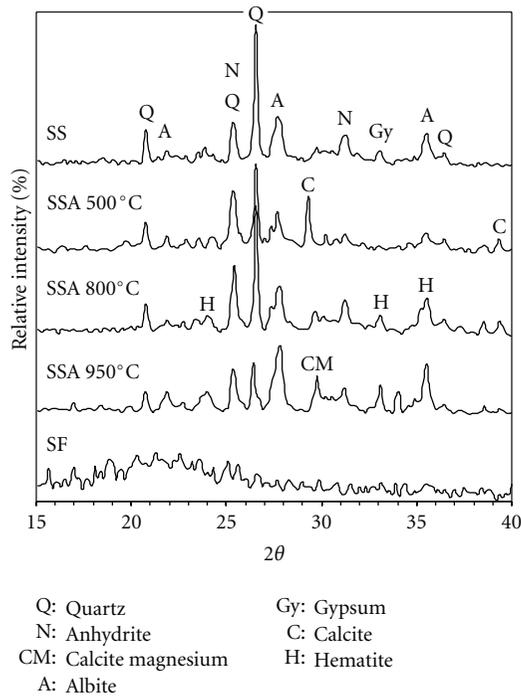


FIGURE 2: The XRD patterns of silica fume, raw sewage sludge, and sewage sludge incinerated at 500–950°C.

concrete [21], lightweight and heat-insulating material [22], ceramic tiles [23], ecocement [24], and for soil stabilization [25]. Attention must be paid to the environmental impacts of sewage sludge ash when it is reused. Principal characteristics must be taken into account to use sewage sludge ash correctly in cement-based materials [26]. It was concluded that the leaching toxicity of sewage sludge ash blended cement is far lower than that of the national standards and minimum harmful matters can be contained and released in the environment [27]. Even sewage sludge ash containing high sulphate content is found to be compatible with cements with high  $C_3A$  content as binder in mortars [28].

Few researchers have indicated that sewage sludge ash has high pozzolanic activity and improves the workability and compressive strength of concrete [29]. On contrast, many researchers have shown that sewage sludge ash has low pozzolanic activity [30] and reduces the workability and

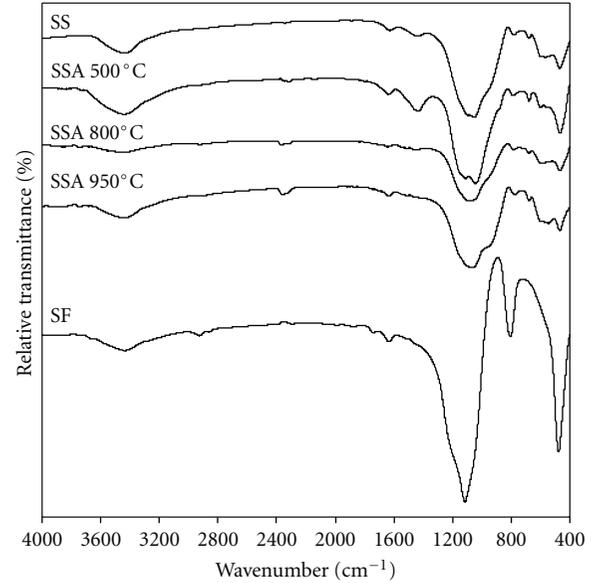


FIGURE 3: The FTIR spectra of silica fume, raw sewage sludge, and sewage sludge incinerated at 500–950°C.

compressive strength of cementitious materials [31]. Incineration of sewage sludge at 600–1000°C greatly affects the chemical composition of the resultant ash [32]. Accordingly, the pozzolanic activity depends on the incineration temperature. Hence, incineration of sewage sludge at different temperatures such as 700°C [33], 800°C [34], or 850°C [26] may lead to the formation of high [35], moderate [36], or less [37] reactive sewage sludge ash and as a result enhances or reduces the workability of cementitious material. Hence, the aim of this study is to clarify the effect of incineration temperature on the microstructure and pozzolanicity of sewage sludge ash.

## 2. Materials and Experimental Techniques

Raw materials used in this study were raw sewage sludge from wastewater treatment facility in Minia Governorate, Egypt and silica fume from the Ferrosilicon Company, Edfo, Egypt. Raw sewage sludge was incinerated in an electrical muffle furnace with a heating rate 10°C/min at 500–950°C for 2 hrs, recharged from the muffle furnace, cooled to room temperature in desiccator, and ground to pass 90  $\mu\text{m}$  sieve. CaO was prepared from pure  $\text{CaCO}_3$  natural powder after incineration at 1000°C, ground and stored in tightly closed plastic bag to avoid carbonation. The pozzolanic activity of incinerated sewage sludge ash and silica fume was evaluated according to Chapelle test. One gram both of sample and CaO were added in 200 mL of distilled water. The mixture was hydrothermally treated at 100°C for 16 h in a stainless steel capsule of 350 mL maximum capacity. Then, the solution was filtrated and the residue was dried. The solution was titrated with 0.1 N HCl solution using phenolphthalein as indicator. Fixed lime content (mmole/L) was calculated from the difference between the amount of

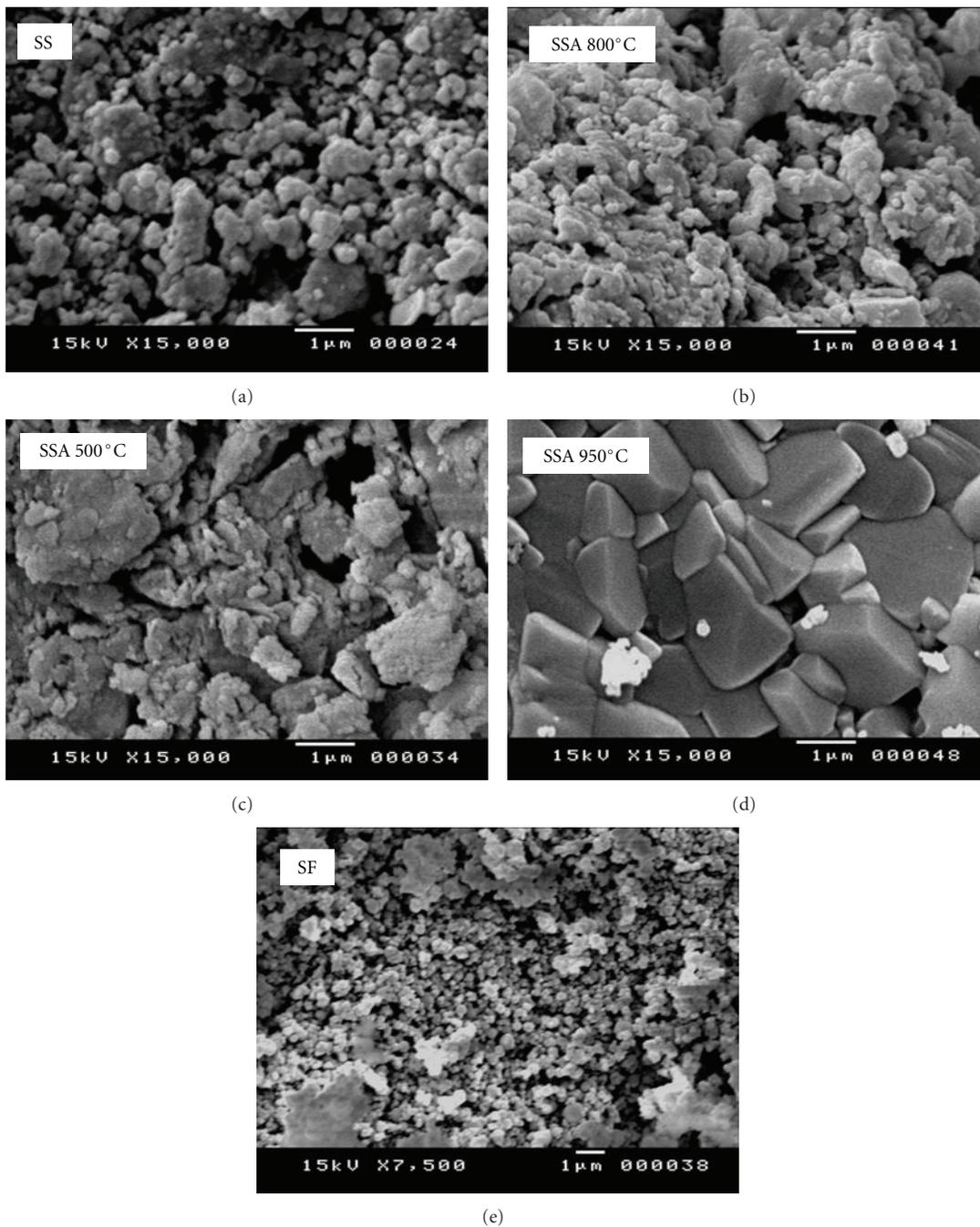


FIGURE 4: The SEM micrographs of silica fume, raw sewage sludge, and sewage sludge incinerated at 500–950°C.

TABLE 1: Chemical composition wt.% of sewage sludge ash incinerated at 800°C and silica fume determined by XRF analysis.

Material	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	LOI*	P <sub>2</sub> O <sub>5</sub>	Total
Sewage sludge ash	39.03	15.13	17.05	5.80	1.93	4.04	0.43	0.62	2.11	13.12	99.26
Silica fume	94.64	0.97	0.93	0.55	0.35	0.10	0.20	0.25	1.65	0.04	99.64

\*LOI is the loss on ignition.

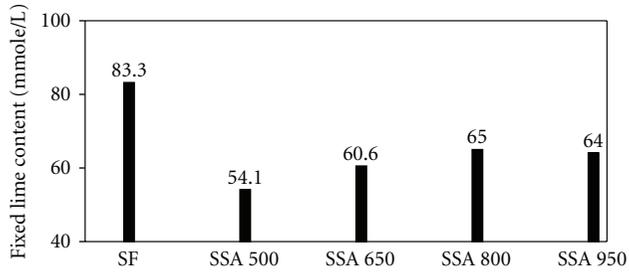


FIGURE 5: The fixed lime content of silica fume and sewage sludge incinerated at 500–950°C hydrothermally treated with lime at 100°C for 16 hrs.

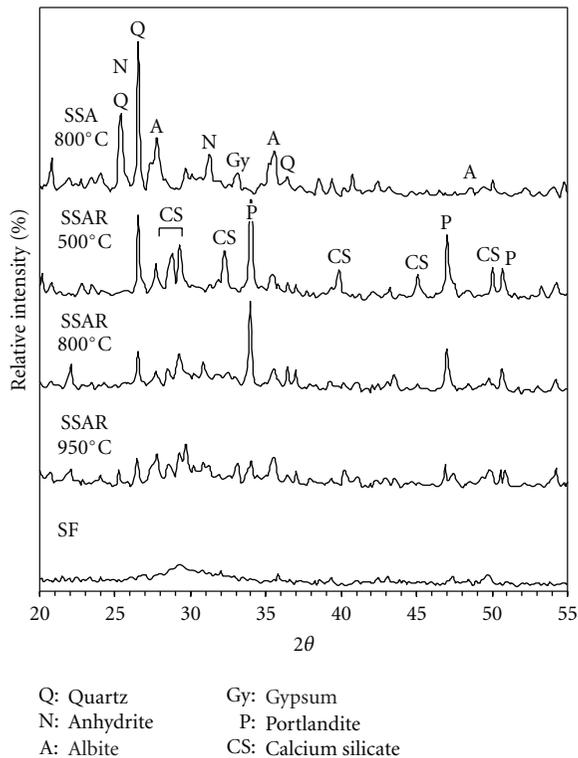


FIGURE 6: The XRD patterns of silica fume and sewage sludge incinerated at 500–950°C hydrothermally treated with lime at 100°C for 16 hrs.

$\text{Ca}(\text{OH})_2$  measured in absence (blank solution) and presence (sample solution) of sample [38].

X-ray fluorescence XRF and X-ray diffraction XRD analyses were carried out by Philips X-ray diffractometer PW 1370, Co. with Ni filtered  $\text{CuK}\alpha$  radiation (1.5406 Å). The Fourier transform infrared FTIR analysis was measured by spectrometer Perkin Elmer FTIR System Spectrum X in the range 400–4000  $\text{cm}^{-1}$  with spectral resolution of 1  $\text{cm}^{-1}$ . Scanning electron microscopy SEM was investigated by Jeol-Dsm 5400 LG apparatus. The thermogravimetric TGA and differential thermogravimetric analyses DTG were carried out with the aid of Shimadzu Corporation thermo analyzer with DTG-60H detector with 10°C/min heating rate from room temperature up to 1000°C, under nitrogen atmosphere

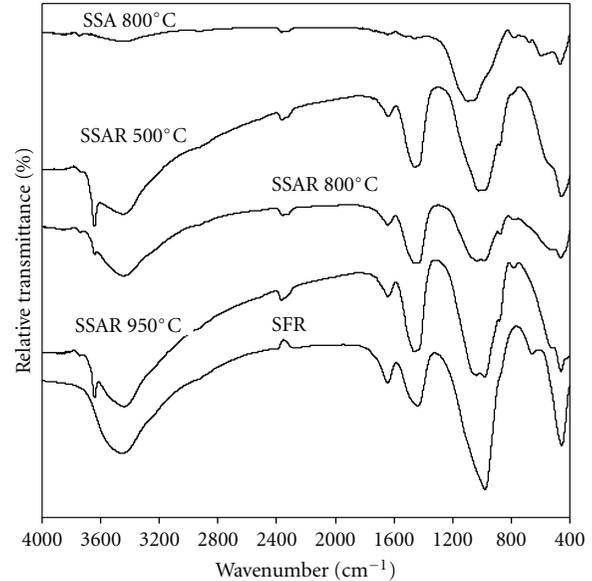


FIGURE 7: The FTIR spectra of silica fume and sewage sludge incinerated at 500–950°C hydrothermally treated with lime at 100°C for 16 hrs.

at 40 mL/min flow rate, the hold time at the appropriate temperature is zero.

### 3. Results and Discussion

**3.1. Characterization of Sewage Sludge Ash.** Table 1 illustrates the chemical composition of sewage sludge ash incinerated at 800°C and silica fume determined by XRF analysis. The sum of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{Fe}_2\text{O}_3$  content in sewage sludge ash is higher than requirements stated in ASTM designation for natural pozzolana [39]. Sewage sludge ash has high  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$  and  $\text{CaO}$  contents due to the use of alum, ferric salts, and lime in wastewater treatment. High percentage of  $\text{P}_2\text{O}_5$  is possibly due to domestic detergents. LOI is possibly due to incomplete incineration and adsorbed water. Silica fume is composed mainly of  $\text{SiO}_2$ .

Figure 1 illustrates the TGA and DTG thermograms of raw sewage sludge. Weight loss which occurs in temperature range 50–130°C is due to elimination of moisture and absorbed water. Weight losses which occur in temperature range 200–320°C and 330–390°C are due to emission of volatile organic matter and combustion of complex non-volatile organic matter, respectively. This is because sewage sludge composes of various species of hydrocarbons with a wide range in boiling point [40]. Weight loss which occurs in temperature range 400–670°C is due to combustion of fixed carbon captured by inorganic materials, elimination of structural water of the clay mineral, and decomposition of the carbonaceous matter present in sewage sludge.

Figure 2 illustrates the XRD patterns of silica fume, raw sewage sludge, and sewage sludge incinerated at 500–950°C. Raw sewage sludge contains quartz  $\text{SiO}_2$ , anhydrite  $\text{CaSO}_4$ , gypsum  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ , and albite  $\text{NaCaSi}_4\text{Al}_4\text{O}_8$  minerals. Calcite  $\text{CaCO}_3$  appears after incineration of sewage sludge

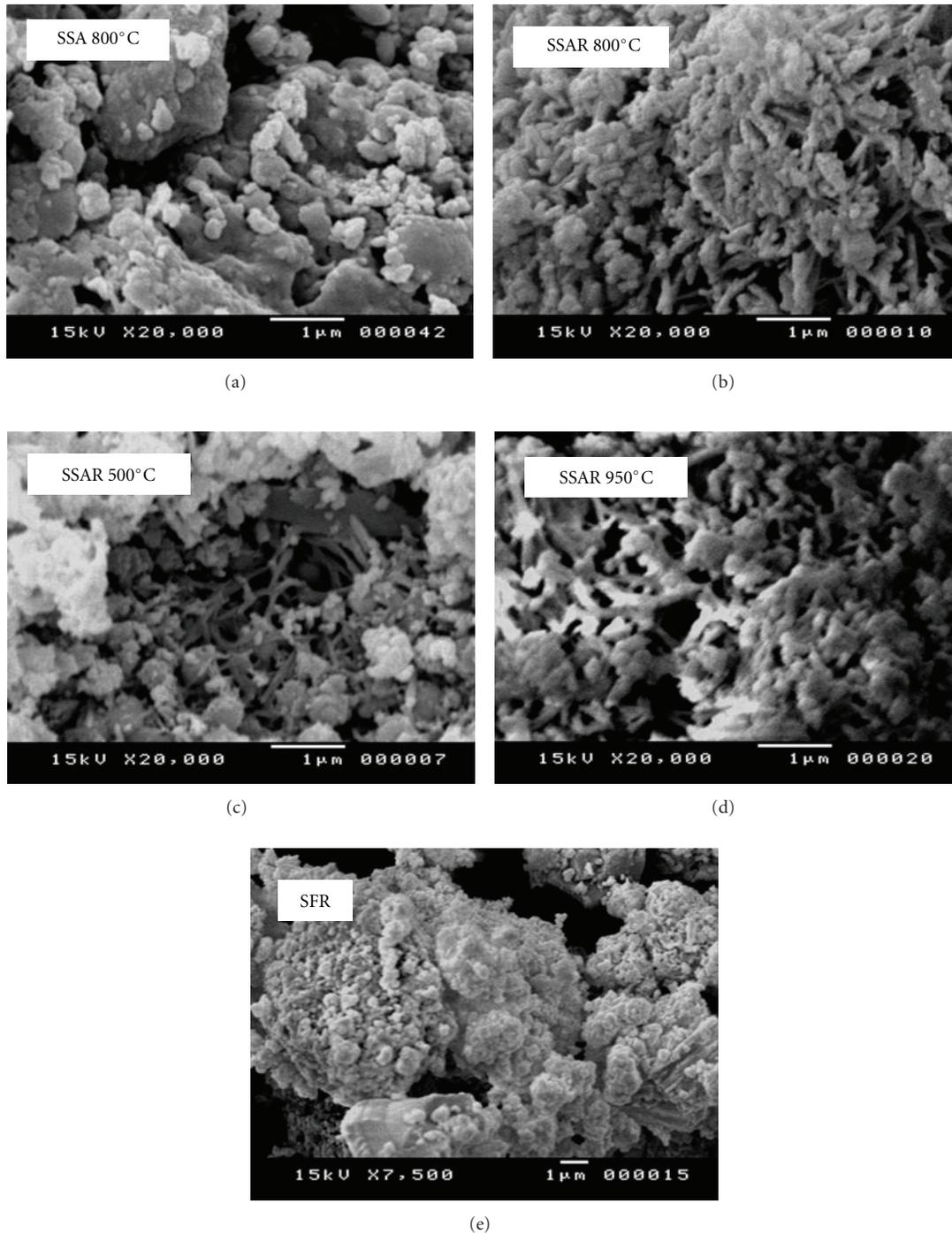


FIGURE 8: The SEM micrographs of silica fume and sewage sludge incinerated at 500–950°C hydrothermally treated with lime at 100°C for 16 hrs.

at 500°C. This may be due to carbonation of some calcium compounds by  $\text{CO}_2$  produced from combustion of organic matter. After incineration of sewage sludge at 800 and 950°C, calcite decomposes into calcium oxide  $\text{CaO}$ . Hematite appears as a result of oxidation of iron compounds. Silica fume is composed mainly from amorphous silica as indicated from the heap in the range 15–30  $2\theta$ .

Figure 3 illustrates the FTIR spectra of silica fume, raw sewage sludge, and sewage sludge incinerated at 500–950°C. The absorption bands of organic matter appear at 681, 1200 and 1628  $\text{cm}^{-1}$ . The absorption band of P-containing compounds appears at 567  $\text{cm}^{-1}$  due to P-O bending vibration [41]. Silica absorption bands appear at 469, 785 and 1052  $\text{cm}^{-1}$  [42]. The absorption band of carbonate appears at

1442  $\text{cm}^{-1}$  [43]. After incineration at 500°C, the absorption bands of organic matter disappear while that of carbonate appears due to carbonation of some calcium compounds by  $\text{CO}_2$  produced from incomplete combustion of organic matter as emphasized from XRD results. Incineration of sewage sludge at 800°C leads to a decrease in the intensity of the absorption bands of silica. This is an indication of increasing the degree of polymerization of silica network as a result of the crystallization of silica into quartz. The intensity of absorption bands of silica increases in case of sewage sludge incinerated at 950°C. This indicates that incineration of sewage sludge at temperature higher than 800°C enhances the crystallization of amorphous silica and this may adversely affect the pozzolanic activity of the resultant ash. Silica fume has absorption bands of silica with high intensity. This proves that silica fume is composed mainly from amorphous silica as emphasized by XRD results.

Figure 4 illustrates the SEM micrographs of silica fume, raw sewage sludge and sewage sludge incinerated at 500–950°C. The organic matters which appear as small aggregates in case of sewage sludge disappear after incineration at 500°C, leaving behind inorganic matter which appear as whitish aggregates. Incineration of sewage sludge at 800°C preserves the amorphous nature of the resultant ash due to elimination of fixed carbon captured by silica. Incineration of sewage sludge at temperatures higher than 800°C enhances the crystallization of amorphous silica. Accordingly, the pozzolanic activity of incinerated sewage sludge may be reduced when sewage sludge incinerated at temperature higher than 800°C. The SEM micrograph of silica fume illustrates that silica fume is mainly composed of amorphous micro silica particles with very high surface area compared with sewage sludge ash. This may prove that the pozzolanic activity of silica fume is higher than sewage sludge ash.

**3.2. Pozzolanic Activity of Sewage Sludge Ash.** Figure 5 illustrates the fixed lime content of silica fume and sewage sludge incinerated at 500–950°C hydrothermally treated with lime at 100°C for 16 hrs. The fixation of lime on sewage sludge ash may occur both by the pozzolanic activity and lime adsorption capacity. The fixed lime content varies with incineration temperature of sewage sludge. Incineration affects the microstructure and amorphous silica content of incinerated sewage sludge. The fixed lime content increases to maximum value at 800°C then slightly reduces at 950°C. This indicates that incineration of sewage sludge at 800°C may be preferred to preserve the pozzolanic activity of the resultant ash. From economic point of view, it is recommended that incineration of sewage sludge at 800°C is better than at higher temperatures. Fixed lime content of silica fume is much higher than that of sewage sludge ash. This reflects the higher pozzolanic activity of silica fume which is composed of amorphous microsilica particles having very high surface area.

Figure 6 illustrates the XRD patterns of silica fume and sewage sludge incinerated at 500–950°C hydrothermally

treated with lime at 100°C for 16 hrs. The amount of portlandite decreases to minimum after incineration of sewage sludge at 800°C, then it increases after incineration at 950°C. While that of calcium silicate varies in an opposite manner. This indicates that incineration of sewage sludge affects its pozzolanic activity. The pozzolanic activity was enhanced with incineration of sewage sludge at 800°C. The pozzolanic activity of sewage sludge ash prepared at 950°C decreases may be due to increasing the degree of crystallization of amorphous silica as emphasized from FTIR and SEM results of sewage sludge ash (Figures 3 and 4). The portlandite was disappeared in case of silica fume hydrothermally treated with lime due to the high pozzolanic activity of silica fume when compared with sewage sludge ash.

Figure 7 illustrates the FTIR spectra of silica fume and sewage sludge incinerated at 500–950°C hydrothermally treated with lime at 100°C for 16 hrs. The absorption band of C–S–H appears at 970  $\text{cm}^{-1}$  due to Si–O stretching vibration [44]. The absorption band at 3640  $\text{cm}^{-1}$  due to portlandite decreases to minimum at 800°C then increases at 950°C. This indicates that the pozzolanic activity of sewage sludge ash prepared at 800°C is higher than at higher temperatures as emphasized from XRD results (Figure 6). The absorption band of portlandite was completely disappeared in case of silica fume hydrothermally treated with lime due to its high pozzolanic activity when compared to sewage sludge ash. The absorption band of carbonate at 1463  $\text{cm}^{-1}$  may be due to partial carbonation of portlandite by atmospheric  $\text{CO}_2$ .

Figure 8 illustrates the SEM micrographs of silica fume and sewage sludge incinerated at 500–950°C hydrothermally treated with lime at 100°C for 16 hrs. The hydrothermal treatment of sewage sludge ash with lime leads to formation of fibrous hydration products. The density of fibers increases to maximum at 800°C then decreases after that. This result is in a good agreement with XRD and FTIR results. This emphasizes that incineration of sewage sludge at 800°C is preferred to preserve the pozzolanic activity of the resultant ash. The morphology of hydration products formed from the hydrothermal treatment of silica fume with lime is different than that formed by sewage sludge ash (i.e., amorphous not fibrous).

Figure 9 illustrates the TGA thermograms of silica fume and sewage sludge incinerated at 500–950°C hydrothermally treated with lime at 100°C for 16 hrs. Weight losses corresponding to the decomposition of C–S–H at 125–350°C are different for all samples [45]. The descending order of these weight losses are silica fume > sludge ash incinerated at 800°C > at 950°C > at 650°C. This indicates that the pozzolanic activity of silica fume is much higher than sewage sludge ash because the former is mainly composed of very fine amorphous silica particles. Sewage sludge ash incinerated at 800°C has high pozzolanic activity and adsorption capacity compared to other sewage sludge ash samples. Incineration of sewage sludge up to 800°C activates the pozzolanicity of the resultant ash. Incineration of sewage sludge at higher temperature enhances the crystallization of amorphous silica and reduces the pozzolanic activity as well as the adsorption capacity of the resultant ash.

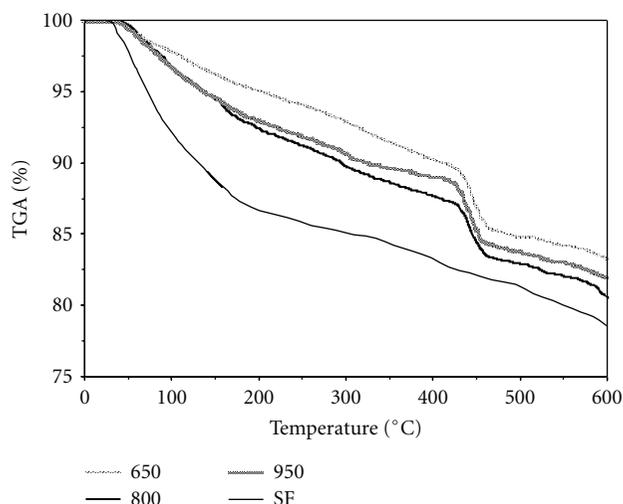


FIGURE 9: The TGA thermograms of silica fume and sewage sludge incinerated at 500–950°C hydrothermally treated with lime at 100°C for 16 hrs.

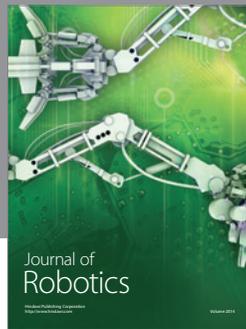
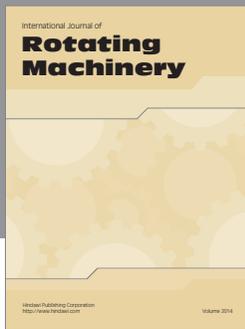
#### 4. Conclusions

The main conclusions of this study are raw sewage sludge contains quartz, anhydrite, and albite minerals while silica fume contains amorphous micro silica. Incineration of sewage sludge at 500–950°C affects the microstructure and pozzolanic activity of the resultant ash. Incineration of sewage sludge at temperature lower than 800°C leads to a decrease in the pozzolanic activity of sewage sludge ash due to that amorphous silica captures fixed carbon resulting from incomplete combustion conditions. Incineration of sewage sludge at temperature higher than 800°C leads to a decrease in the pozzolanic activity of sewage sludge ash due to crystallization of amorphous silica. Hence, incineration of sewage sludge ash must be optimized at 800°C to preserve the pozzolanic activity of the resultant ash. Hydration products formed from hydrothermal treatment of silica fume with lime is amorphous whereas that of sewage sludge ash is fibrous.

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